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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.

10/830,208

Cnfrm. No.

9026

Applicant(s)

Todd A. Leonhardt et al.

Filed

22-APR-2004

Title

BINARY RHENIUM ALLOYS

TC/A.U.

1742

Examiner

Ngoclan Thi Mai

Docket No.

RHE-15250

Honorable Commissioner for Patents Alexandria, VA 22313-1450

DECLARATION OF TODD A. LEONHARDT UNDER 37 C.F.R. §1.132

Sir:

The undersigned, Todd A. Leonhardt, hereby declares that:

- 1. Declarant is one of the joint inventors in the above-referenced application.
- 2. Declarant is presently employed as Director of Technology of Rhenium Alloys, Inc., a corporation organized under the laws of the State of Ohio having a place of business at 1329 Taylor Street, Elyria, Ohio 44036-0245.
- 3. Declarant earned a Bachelor of Science degree in Chemistry from Baldwin Wallace College and an Associate of Science degree in Metallurgy from Pennsylvania

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State University.

- 4. Declarant has 23 years of experience in the technical fields of metallurgy and chemistry.
- 5. Declarant has significant experience working with rhenium and with various alloys containing rhenium. Declarant has authored and presented at least 12 technical papers concerning the processing and use of rhenium and its alloys, and is frequently requested to lecture on the subject at seminars.
- 6. Declarant is familiar with the process for the preparation of refractory metal powders described in Foster, Jr. et al., U.S. Pat. No. 3,318,683 (hereinafter the "Foster Jr. et al. patent"). In declarant's opinion, the process described in the Foster Jr. et al. patent is not a commercially viable process because it is prohibitively expensive. It is declarant's understanding and belief that the process described in the Foster Jr. et al. patent has only been practiced in a laboratory setting.
- 7. The Foster, Jr. et al. patent describes in Example 3 (col. 5, line 65 to col. 6, line 2) the production of an alloy consisting of 90% by weight rhenium and 10% by weight tungsten as follows:

A 40-gram alloy of Re-10 w/o W is melted in an electric are furnace at 3200° C. in an atmosphere of ammonia whose pressure is maintained at 2 atmospheres. When gas equilibrium is established at a level where 15 p.p.m. of nitrogen are dissolved in the molten metal, the metal is rapidly cooled to room temperature in about two minutes.

The solid metal is ground to a powder in a ball mill and the residual nitrogen in the powdered metal is removed in a vacuum oven at a temperature of 1200° C. under vacuum. The powdered nitrogen free metal is then ready for use in a very pure state.

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- 8. In 1964, when the application that became the Foster Jr. et al. patent was filed, it would have been difficult to produce fine refractory metal powders. The Foster Jr. et al. patent refers to the final product as being in a "very pure state", which would be true by 1964 standards, but is not true by today's standards. By today's standards, the metal powder produced according to the process described in the Foster Jr. et al. patent would not be considered high purity material due to the contamination inherently introduced into the material by melting and ball milling.
- 9. The Foster Jr. et al. patent states at col. 3, lines 8-13 that the metals (e.g., Re, W etc.) were melted in "electric arc furnaces". This equipment uses a copper cooled wall. When producing rhenium-based alloys, copper is a significant contaminant, which causes liquid metal embrittlerment. Embrittlement is one of the objects of the Foster Jr. et al. patent because the powder is produced by ball milling the metal.
- 10. Example 3 of the Foster Jr. et al. patent states that metals were melted in an electric arc furnace in an atmosphere of ammonia. The ammonia was cracked ammonium, which is a poor reducing agent and results in the production of a final product having a relatively high nitrogen and oxygen concentration. Because Example 3 used a gas having a limited reducing capability, the product likely contained a high oxygen and nitrogen concentration (at least as compared to that of alloys formed in accordance with applicants' claimed process), which further produces brittleness in the metal. Again, this is one of the objects of the Foster Jr. et al. patent because embrittlement allows for the production of fine particles by ball milling. The introduction of brittleness into the alloy would significantly reduce its strength and ductility.

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- 11. On information and belief, an alloy produced as described in Example 3 of Foster, Jr., et al., would not exhibit a room temperature tensile strength in excess of 150 ksi and an elongation of 25% or greater as measured in accordance with ASTM E8-03. As noted in paragraph [0029] of the present application, alloys of refractory metals that are formed by electric arc melting the constituent metals together exhibit a grain structure that is substantially larger than the grain structure of alloys formed according to the process described in the above-referenced application. This difference in grain size is observed regardless of the rate at which the molten metal is cooled. Furthermore, it is likely that the alloy formed according to the process described in the Foster Jr. et al. patent would exhibit porosity due to entrapped gas, which significantly reduces the properties of the alloy. Furthermore, as noted in the same paragraph of the present application, it is very difficult to obtain a good dispersion of the tungsten throughout the bulk of the rhenium when the two constituent metals are arc melted together. Rhenium-tungsten alloys produced according to the method described in the present application exhibit an unexpected a room temperature tensile strength and an elongation percentage that cannot be obtained by arc melting the constituent metals together to form a uniform alloy.
- 12. Examples 7 and 10 of the Foster, Jr., et al. patent describe the production of rhenium alloyed with iridium and osmium, respectively. Again, inasmuch as these alloys are formed by arc melting the constituent metals together, on information and belief, such alloys would exhibit a substantially larger grain structure than alloys having the same weight percent composition formed according to the process described in the present application. This difference in grain structure, as well as differences between

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the uniformity of the dispersion of the alloying metals through the bulk of the rhenium, would result in the production of alloys having significantly different mechanical properties. Upon information and belief, alloys produced according to the process disclosed in the present application would exhibit substantially greater room temperature tensile strength and elongation percentage than alloys formed according to the process described in Foster, Jr. et al.

- 13. Declarant is also familiar with the process of forming a tungsten electrode rod for a high-pressure metal halide discharge lamp described in Kubon et al., U.S. Pat. No. 6,060,829 (hereinafter "the Kubon et al. patent"). At col. 3, lines 22-37, the Kubon et al. patent teaches that a portion of a tungsten electrode rod should be coated with rhenium, or an alloy of rhenium containing no more than 10% by weight (or more preferably no more than 5% by weight) of tungsten. The Kubon et al. patent teaches that the rhenium or rhenium-tungsten alloy can be coated on the tungsten electrode by wrapping with a wire, or a foil, or by depositing rhenium or a tungsten-rhenium mixture onto the tungsten electrode by sputtering or by vapor deposition. Obviously, this would not produce a homogenous alloy, but rather a tungsten rod coated with a rhenium or rhenium alloy coating.
- 14. The Kubon et al. patent does not describe how the "wire" of rhenium or an alloy of rhenium and tungsten is formed. At the time the priority application that became the Kubon et al. patent was filed in the European Patent Office on February 24, 1997, the only commercially available rhenium-tungsten alloy wire available was produced by powder metallurgy process. This commercially rhenium-tungsten alloy wire produced was by diffusion of tungsten and rhenium. This commercially available wire did not

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exhibit a room temperature tensile strength in excess of 150 ksi and an elongation of 25% or greater as measured in accordance with ASTM E8-03.

- 15. Declarant is also familiar with the process of forming a furnace component having a tungsten-based substrate and a surface protected with a rhenium-based layer described in Bewlay et al., U.S. Pat. No. 6,162,552 (hereinafter "the Bewlay et al. patent"). Similar to the process described in the Kubon et al. patent, a wire of rhenium or an alloy of rhenium and up to about 11% by weight tungsten was closely wound on a tungsten-based substrate and then heat treated to sinter the rhenium or rhenium-based wire to the substrate (see col. 3, line 57 to col. 4, line 30). This is a coating process, and not a solid-state alloy process.
- 16. The Bewlay et al. patent does not describe how the "wire" of rhenium or an alloy of rhenium and tungsten is formed via a layer or deposition or coating process. At the time the Bewlay et al. patent application was filed in the U.S. Patent Office on December 3, 1998, the only commercially available wire of rhenium and tungsten were formed via powder metallurgy processing. The resultant material described by the Bewlay et al. patent was produced via layering process. Upon information and belief, neither the starting material or the resultant material would exhibit the properties at room temperature with tensile strength in excess of 150 ksi and an elongation of 25% or greater as measured in accordance with ASTM E8-03.
- 17. The undersigned declares that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both,

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under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Todd A. Leonhardt

Date Signed: /2/12/05